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A Semi-Automatic Stereometer

Boom, G.; Sikkens, M.; Hosson, J.Th.M. De

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BETWEEN BULK AND SURFACE: THE POSITION OF TEM IN THE CHARACTERIZATION OF CATALYSTS

A. Knoester

*Koninklijke/Shell-Laboratorium,
Amsterdam (Shell Research B.V.),
Badhuisweg 3, 1031 CM Amsterdam*

Insight into the functioning of catalysts requires, among other things, knowledge about the nature of the catalytic components and their interactions, and about the extent, structure and accessibility of the active surface. These properties can be studied by, e.g., surface techniques (such as XPS) and bulk techniques (such as XRD).

TEM can often correlate the observations from these disciplines, thus allowing a more detailed characterization of the surface as well as the bulk of the catalyst. To this end the micrographs are interpreted within the constraints imposed by the other techniques. The success of this multidisciplinary approach is demonstrated with the help of various examples.

HIGH RESOLUTION ELECTRON MICROSCOPY OF CATALYTIC MATERIALS

H.W. Zandbergen

Gorlaeus Laboratories, State University of Leiden, PO Box 9502, 2300 RA Leiden, The Netherlands

High Resolution Transmission Electron Microscopy (HRTEM) is a powerful tool for the investigation of catalytic materials. In the past electron microscopy was used mainly for determination of particle size distributions of small metal particles on support. Furthermore, movement due to sintering of metal particles down to 5 nm were examined in situ.

Since about five years the electron microscopes have been so improved that lattice spacings down to 0.19 nm can be made visible. In the characterization of catalytic materials this is a very essential step forward. With the new generation of electron microscopes lattice images of small metal particles and support can be obtained, giving information on crystallinity, purity and orientation. For instance, HRTEM experiments on Pt on alumina support show that the Pt particles of about 5 nm are crystalline, having the same lattice type as Pt metal.

On the other hand, Heinemann and Poppa reported that Pd particles in the 1-2 nm size range have a crystal structure different from that of Pd metal.

We discuss some of our experiments on Rh and Ir particles on titanium oxide and on zeolites containing small metal particles. HRTEM experiments were carried out on a number of Rh/TiO₂ and Ir/TiO₂ samples to investigate:

- 1) The influence of defocus, objective aperture size and other instrumental parameters on the observed sizes of metal particles;
- 2) Do changes, caused by the electron flux in the electron microscope, occur in the metal particles or the support?
- 3) Can sintering experiments be simulated in the electron microscope using a dense electron flux?
- 4) Does the movement of small metal particles depend on the crystallographic plane of the surface of the support on which they are moving?

The depiction of lattices of zeolites is strongly hindered by their sensitivity to the electron beam—a problem when one wants to know the position of small metal particles inside the zeolite lattice. But this beam sensitivity can be useful. Metal particles can be much better detected when the zeolite lattice is destroyed by the beam. Contrary to the Rh/TiO₂ system, e.g., where besides a sintering of the support a sintering of the metal particles occurs, no differences in particle size are observed before and after destruction of the zeolite lattice. Another useful technique in the depiction of small metal particles is a deliberate misorientation of the zeolite crystal. Various ways of imaging metal particles in zeolites are presented.

MATERIAL SCIENCE AND PHYSICS

A SEMI-AUTOMATIC STEREOMETER

G. Boom, M. Sikkens and
J.Th.M. De Hosson

Vakgroep Technische Fysica, Materials Science Centre, Rijksuniversiteit Groningen, Nijenborgh 18, 9747 AG Groningen, Netherlands

A Hilger & Watts SB190 stereometer modified by Boom was extended by addition of an X-Y mechanism driven by stepping motors via a 1 mm pitch screw-spindle. Since the Philips HR23 motors used in their half-step mode take 400

half-steps per revolution, the minimum translation increment is 2.5 μm . The span is 240 mm in X and Y, parallel and perpendicular to the stereo axis, respectively. This also defines the measurable photo area. The maximum measurable stereoparallax (ΔY) is 50 mm.

The X, Y and ΔY values are read by SONY Magnescale LY-101-12 digital counters from SONY Magnescale magnetic measuring rods which have an absolute position error of 3 μm over their length.

An interface was built to read the Magnescale counters into an Apple II+ personal computer, and another to drive the stepping motors, which can also be driven with a joystick. The parallax measurement is done with the light spots of the SB190 (adjusted to fit the observer's pupil distance) by manually adjusting the ΔY position of the right-hand photo, until the three-dimensional images of both the light spot(s) and selected detail in the photo(s) coincide in X, Y and Z values. X and Y values are divided by the magnification M of the photos, while $Z = [\Delta Y / \sin \phi - Y \tan(\phi/2)] / M$.

These X-Y-Z sets referring to the left-hand photo are stored on disk and may either be transferred to the central University computer for processing and/or graphical display or be read by another Apple program which provides a graphical display in which true distances between two observer-selected points are calculated along with the Miller indices of the line joining these two. The Miller indices of a plane defined by three selected points may be calculated. New projections along any direction can be calculated and displayed. Any display may be printed as it appears on the video screen.

RAMAN MICROSCOPY OF SOIL SAMPLES

G.H.M. Gijsbers, S. Henstra*, E.B.A. Bisdorf** and J. Greve

*Biophysical Technology, Dept. Applied Physics, Twente University of Technology, PO Box 217, 7500 AE Enschede; *Technical and Physical Engineering Research Service, and **Soil Survey Institute, Wageningen; Netherlands*

In Raman microspectroscopy the Raman scattering of a visible laser beam, focussed through a normal light microscope, can be used to identify the molecular composition of the scattering material. Particles of a few micrometers in diame-

ter can thus be analyzed.

The samples used are a thin section of peat and thin sections of sandy soil material with fine-grained and amorphous black and brown organic matter between sand grains. The soil material is embedded in a polyester/polystyrene resin.

In the non-organic particles the presence of α -quartz, anatase and rutile can be observed. In the peat section the Raman spectrum of the organic matter, superimposed on a broad fluorescence emission, indicates the presence of graphitized matter, because the bands at 1600 cm^{-1} and 1355 cm^{-1} can be assigned to small disordered graphite crystals.

The fluorescence of the organic matter in the sandy soil was in most cases too intense to distinguish more than just a hint of any Raman band. The resin could clearly be distinguished from the soil material by its characteristic Raman spectrum.

In the future the problem of the fluorescence which obscures the Raman signal can possibly be solved using CARS (Coherent Anti-Stokes Raman Scattering) microscopy. The possibilities of this method are now under study in our laboratory.

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AUTOFOCUSING AND AUTOALIGNMENT OF A TEM

A.J. Koster, R. Swaan, F.J. Pijper, L.T.M. Heesterbeek and K.D. van der Mast

Research Group for Particle Optics, Department of Applied Physics, Delft University of Technology, PO Box 5046, 2600 GA Delft, The Netherlands

Tilting the illuminating beam in a transmission electron microscope (TEM) gives a displacement of the image of the specimen on the screen when the TEM is out of focus. This displacement has a known relationship with the defocus of the TEM. Autofocussing is possible by having a computer tilt the illuminating beam, measure the displacement of the image, calculate the defocus and correct the defocus. Correction of astigmatism is possible by measuring the defocus in different directions. By measuring the image shift for several tilt angles it is possible to align the illuminating